Jack Kelley & Tom Braswell
jkelley@arl.army.mil & thomas.e.braswell@arl.army.mil

U.S. Army Research Laboratory
BLDG 4600, Deer Creek Loop
APG MD 21005

Luwam Hagos & Amy Fowler
luwam.hagos@navy.mil & amy.fowler1@navy.mil

U.S. Naval Air Warfare Center
NAVAIR, Code 4.3.4.2
48066 Shaw Road, Bldg. 2188
Patuxent River, MD 20670-1906

TECHNOLOGY DRIVEN. WARFIGHTER FOCUSED.

Non-chromate, Zero-VOC Coatings for Steel Substrates on Army and Navy Aircraft and Ground Vehicles

SAN DIEGO CA
August 28-30 2012

TECHNOLOGY DRIVEN. WARFIGHTER FOCUSED.

Report Documentation Page

Form Approved
OMB No. 0704-0188

1. REPORT DATE	2. REPORT TYPE	3. DATES COVERED
AUG 2012		**00-00-2012 to 00-00-2012**

4. TITLE AND SUBTITLE	5a. CONTRACT NUMBER
Non-chromate, Zero-VOC Coatings for Steel Substrates on Army and Navy Aircraft and Ground Vehicles	5b. GRANT NUMBER
	5c. PROGRAM ELEMENT NUMBER
6. AUTHOR(S)	5d. PROJECT NUMBER
	5e. TASK NUMBER
	5f. WORK UNIT NUMBER
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) **U.S. Army Research Laboratory,BLDG 4600, Deer Creek Loop,APG,MD,21005**	8. PERFORMING ORGANIZATION REPORT NUMBER
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)	10. SPONSOR/MONITOR'S ACRONYM(S)
	11. SPONSOR/MONITOR'S REPORT NUMBER(S)

12. DISTRIBUTION/AVAILABILITY STATEMENT
Approved for public release; distribution unlimited

13. SUPPLEMENTARY NOTES
Presented at the ASETSDefense 2012: Workshop on Sustainable Surface Engineering for Aerospace and Defense August 27-30, 2012, San Diego, CA

14. ABSTRACT

15. SUBJECT TERMS

16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON
a. REPORT **unclassified**	b. ABSTRACT **unclassified**	c. THIS PAGE **unclassified**	**Same as Report (SAR)**	**28**	

Project Team Acknowledgements

Name	Organization	Contribution
Jack Kelley--PI Tom Braswell	ARL; Aberdeen, MD	Principal Investigator. Lead coating evaluations and specification testing. Primary POCs for Army demonstration efforts.
Luwam Hagos--PI Amy Fowler	NAVAIR Materials Engineering Pax River, MD	Principal Investigator. Lead coating evaluations and specification testing. Primary POCs for Navy demonstration efforts.
Tom Considine	ARL, Aberdeen	Corrosion Testing
John A. Escarsega	DoD CARC Commodity Manager	Organic Coatings
Patricia Dodson Austin Minter	Anniston Army Depot Anniston AL	Department of Engineering Quality. Coordinate processing of Stryker Demonstrations, and phosphate sealer demonstration
James Swann	PM SBCT Reset ANAD	Reset Coordinator at Stryker Brigade Combat Team. Coordinate Stryker dem/val
Stephen Bails	PM SBCT EMT	Coordination of Stryker demonstration site
Todd Weimer	PMO-MRAP Warren MI	Develop performance requirements for MRAP
Daniel (Dusty) Cooper	Camp Lejeune NC	Coordination of MRAP vehicles and facilities at Camp Lejeune
Jacob Waller	FRC Cherry Point, NC	Depot lead for NAVAIR phosphate sealing
Keith Legg	Rowan Technology Group	Economic and Environmental Impact

ESTCP WP 200906 Project

Total of 3 Technology Areas Being Demonstrated

ESTCP WP 200906
Evaluate Commercially Available Pretreatments and Coatings for Steel Substrates

- **1) Demonstration of Conversion Coatings for Armor Steel**
  - Stryker (ANAD)- Jim Swann/Patty Dodson
  - MRAP (Camp Lejeune)- Daniel Cooper CWO5 (ret)
- **2) Demonstration of Phosphate Sealers**
  - Anniston Army Depot – Patty Dodson
- **3) Demonstration of Non-chromate primer and ZVOC Topcoat**
  - Cherry Point – Jacob Waller

TECHNOLOGY DRIVEN. WARFIGHTER FOCUSED.

Overall Project Objectives

Demonstrate and validate non-chromate, zero-voc coatings on Army and Navy systems and recommend for implementation proven alternatives at military and OEM facilities:

1. Pretreatments for armor steel for more robust paint systems
2. Eliminate chromic acid rinses in zinc phosphate operations
3. Expand the use of zero-voc topcoats on ground support equipment





ESTCP WP 200906 Project

Total of 3 Technology Areas Being Demonstrated

ESTCP WP 200906
Evaluate Commercially Available Pretreatments and Coatings for Steel Substrates

- **1) Demonstration of Conversion Coatings for Armor Steel**
  - Stryker (ANAD)- Jim Swann/Patty Dodson
  - MRAP (Camp Lejeune)- Daniel Cooper CWO5 (ret)
- **2) Demonstration of Phosphate Sealers**
  - Anniston Army Depot – Patty Dodson
- **3) Demonstration of Non-chromate primer and ZVOC Topcoat**
  - Cherry Point – Jacob Waller

TECHNOLOGY DRIVEN. WARFIGHTER FOCUSED.

Background

Chromic Acid Usage at Anniston Army Depot

8oz (1/2 pound) per 100 gallons.
Total chromic acid is 6862 lbs issued in 2011

1 pound = 200 gallons of solution
6862 pounds x 200 gallons = **1,372,400** gallons of solution annually

Objective: Eliminate chromic acid rinses in zinc phosphate operations

Test Methodology

MIL-DTL-16232: Zinc Phosphate

❖ Baseline Sealer:
- •Chromic acid rinse
- •(1 pound = 200 gallons of solution)

❖ “Drop in” Replacement Candidates:

PPG Chemseal 100
- • Non-chrome post-rinse with an inorganic hexafluorometalate component and an organic resin component.
- • Makes the phosphate layer less susceptible to alkaline dissolution and promotes adhesion.

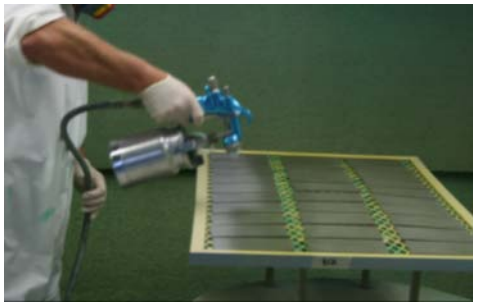
SurTec 580 ChromiPhos
- •Trivalent chrome passivation for phosphating
- • Forms protective layer of metal, complex oxides and hydroxides on exposed surface

❖ Tested under three paint systems:
- •MIL-DTL-53022/ MIL-DTL-53039
- •MIL-DTL-53022/MIL-DTL-64159
- •MIL-DTL-23377/MIL-PRF-85285 ZVOC

Test Methodology

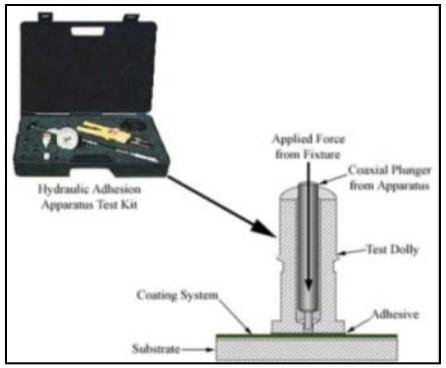
Substrates:

- Low Carbon Steel A366 (Milled Finish)
- High Hard Armor MIL-A-46100 (Abrasive Blasted)
  - Charpys
- *All pretreatments applied by manufacturer*



Adhesion Test:

- Pull-Off Adhesion – ASTM D 4541
- Wet Adhesion – ASTM D 3359 Method A



Paint Adhesion

Accelerated Corrosion Chamber

Corrosion Tests:

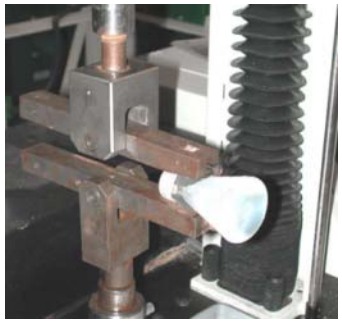
- Neutral Salt Fog – B117
- Cyclic Corrosion – GM9540P
- Outdoor Exposure (Cape Canaveral)

Chip Resistance:

- Gravelometer – SAE-J400

Stress Corrosion Cracking:

- Rising Step Load – ASTM F 1624-95



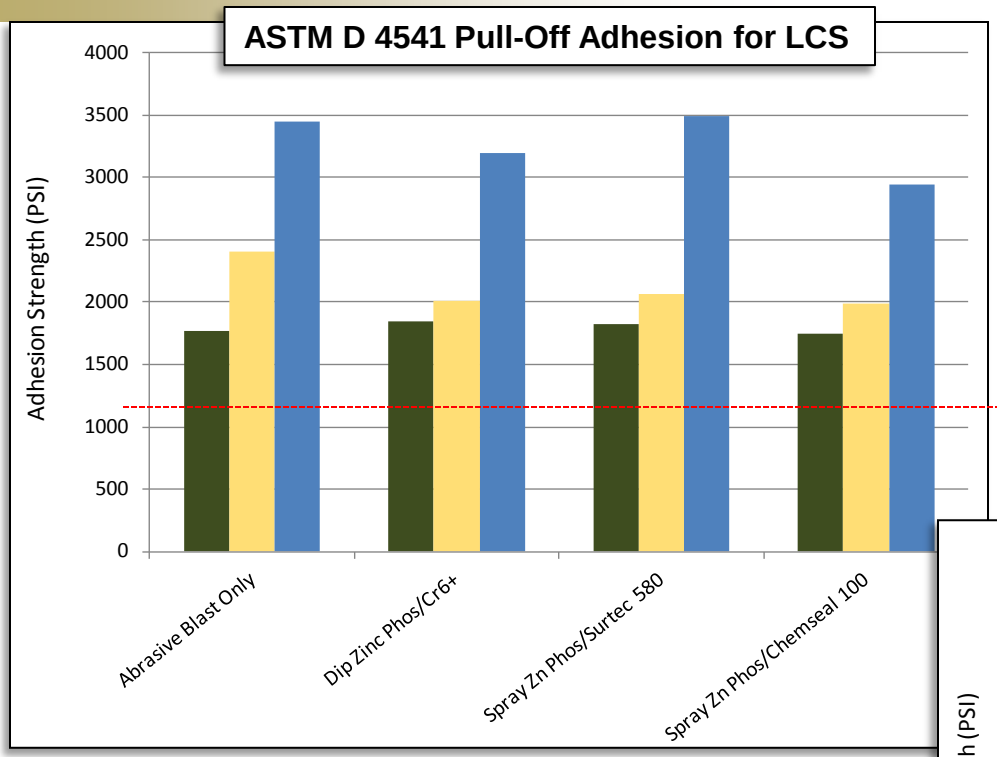
Rising Step Load

Test Methodology

Quantitative Performance Objectives

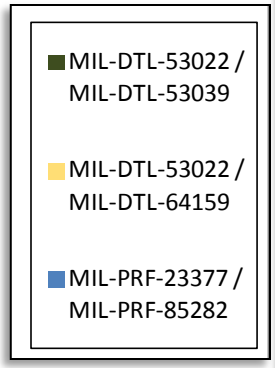
Performance Objective	Data Requirements	Success Criteria
Adhesion Test	ASTM-4541 Pull-off Adhesion ASTM- D3359 Dry Adhesion ASTM- D3359 Wet Adhesion	Minimum average 30 events rating of **1200 PSI on 1.5 mil profile surface** Adhesion rating (steel) > 4B; adhesion rating Scribed area rating (steel) ≥ 3A after 24 hours at ambient;
Chip Resistance	SAE-J400	**NLT 5B**
Accelerated corrosion	ASTM-B117 Salt Fog	After 500 hours of exposure: steel substrate rating **≥6 scribed**
	GM-9540P Cyclic Corrosion ASTM D 1654	After 60 cycles: steel substrate **rating ≥ 4**
Outdoor Exposure	Tropical climate exposure at Kennedy Space Center Outdoor Site. ASTM D 1654 ASTM G50	Three years of exposure: Equivalent or less average creepage from scribe than current corrosion protection system
Processing time	TT-C-490	Equivalent or less than existing process
Field Testing	SSPC-VIS-2 (more details in section 5.0)	Equivalent or less than existing process
Hydrogen Embrittlement	ASTM F 1624-09 ASTM E 399-97	No detrimental effect to K1c of substrate. High Hard K1c @ 48-51Rc shall maintain K1eac ≥ 19 (ksi√in)

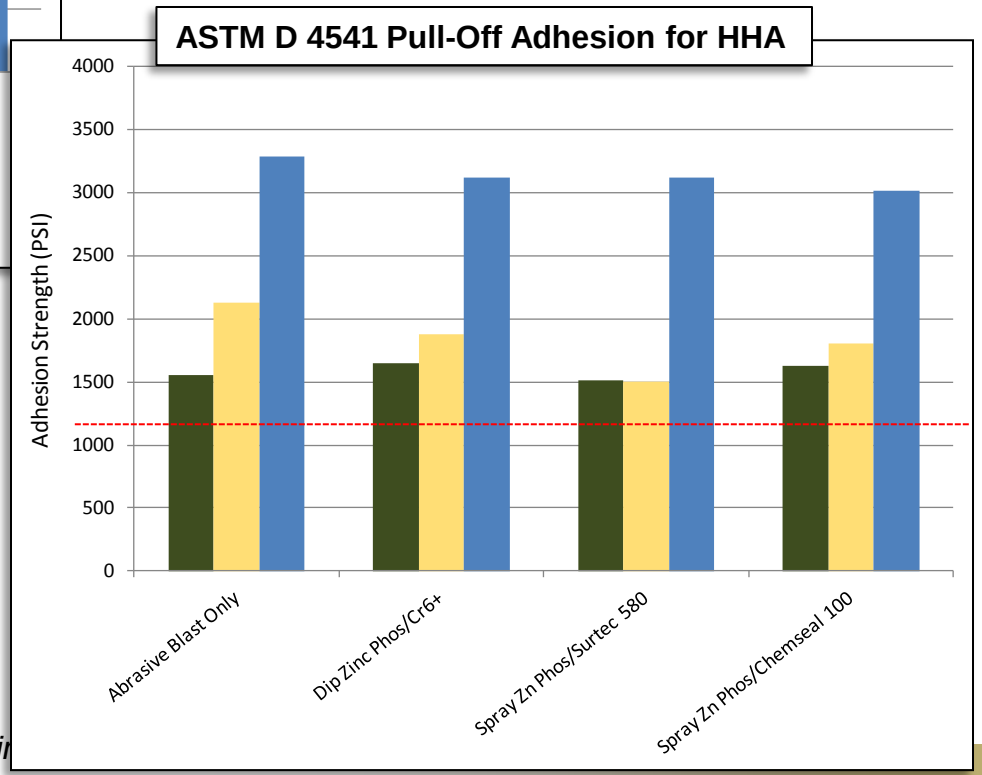
Laboratory Validation Results



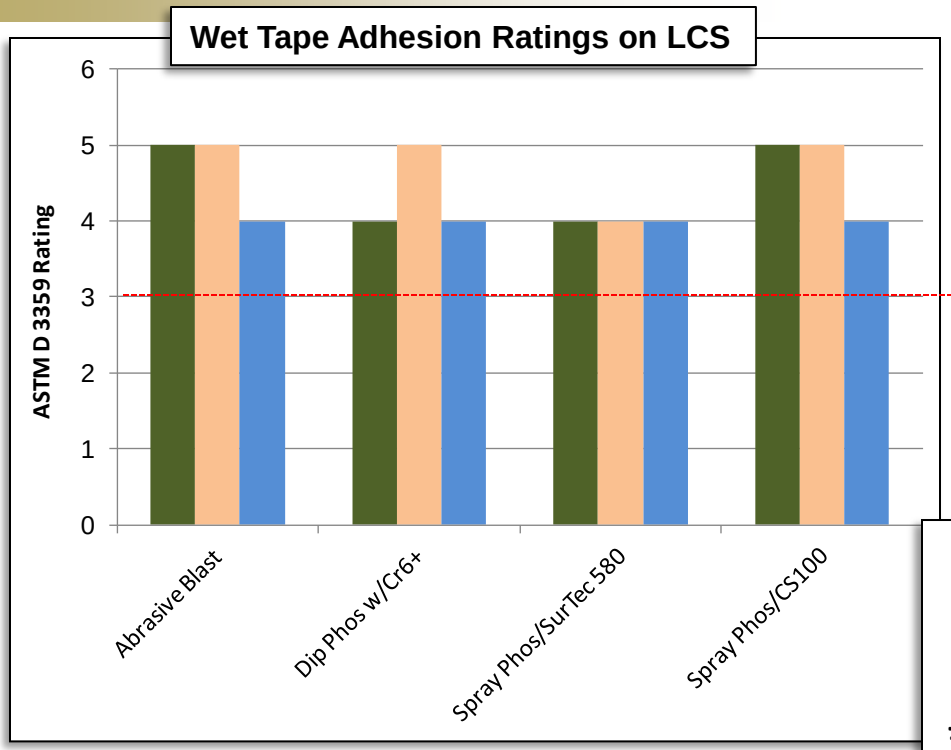
ASTM D 4541 results for zinc phosphated steel panels

- Pull off values for all considered artificially low on CARC Beaded versions.
  - Suspected glue/bead interaction





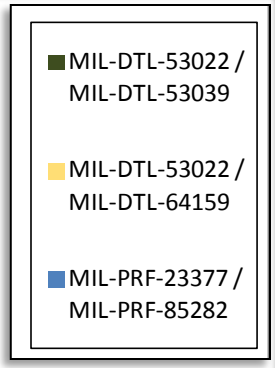
Laboratory Validation Results

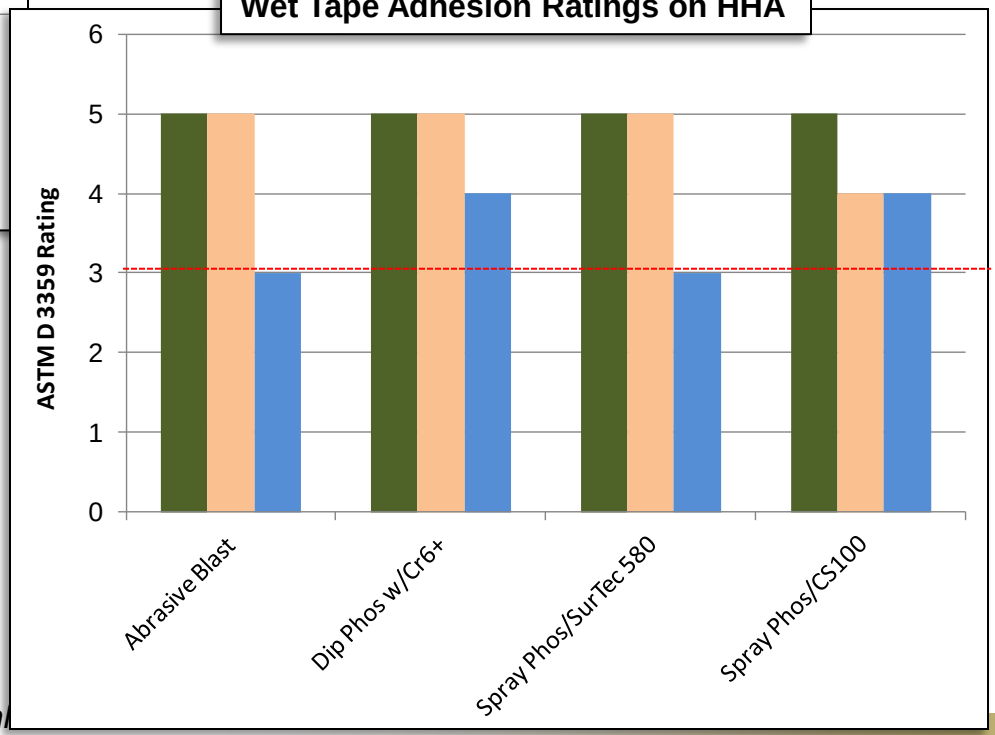


Wet tape adhesion results for pretreated steel panels

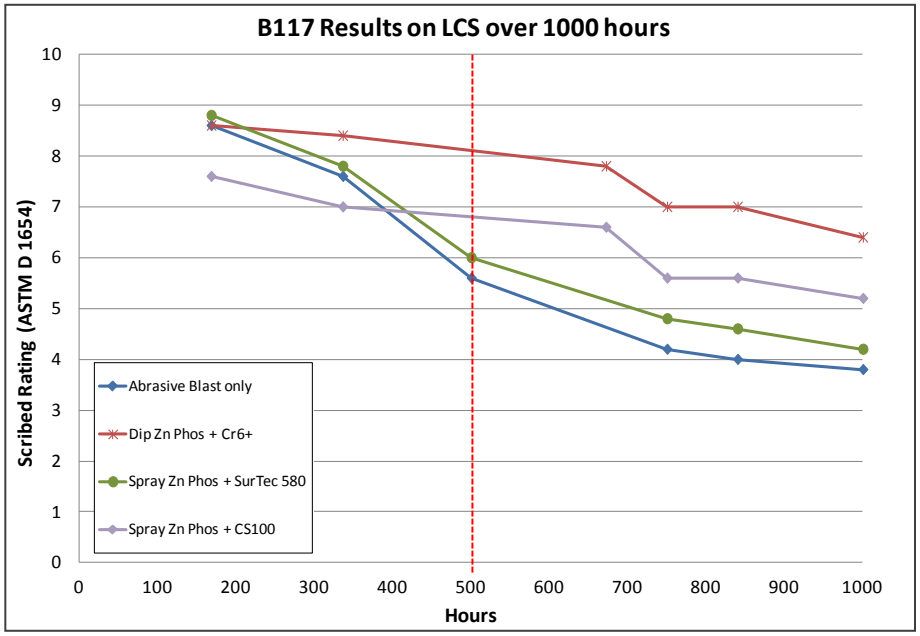
- Ratings per ASTM D 3359
- 24 hour immersion in DI water

Cross-hatch tape pull



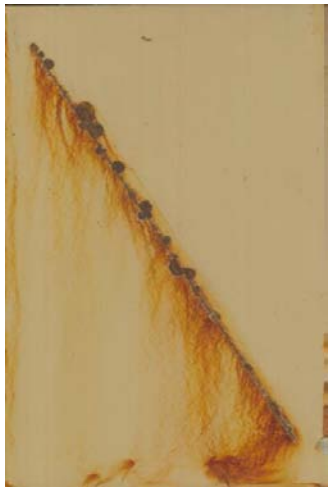


Laboratory Validation Results



- Phosphated low carbon steel panels
- Coatings system 53022/53039 CARC system
- ASTM-B117 results over 1000 hours of exposure
- Rated using ASTM D1654

Dip Zn Phos/Cr6+

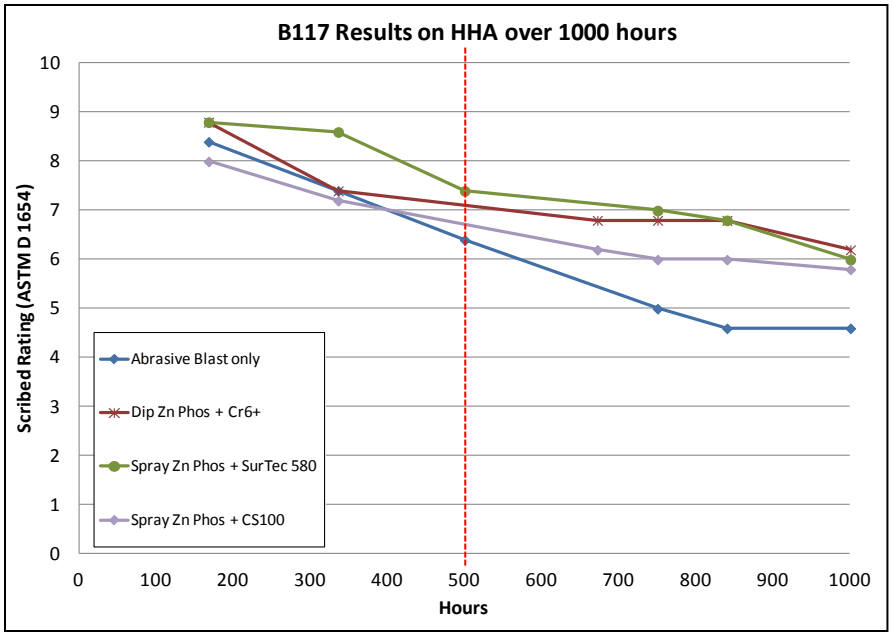


Spray Zn Phos/SurTec 580

Spray Zn Phos/PPG Chemseal 100

TECHNOLOGY DRIVEN. WARFIGHTER FOCUSED.

Laboratory Validation Results



- Phosphated HHA steel panels
- Coatings system 53022/53039 CARC system
- ASTM-B117 results over 1000 hours of exposure
- Rated using ASTM D1654

Dip Zn Phos/Cr6+	Spray Zn Phos/SurTec 580	Spray Zn Phos/PPG Chemseal 100

TECHNOLOGY DRIVEN. WARFIGHTER FOCUSED.

Laboratory Validation Results

ASTM-D1654 Ratings for Low Carbon Steel (LCS) with 53022/53039						
		GM 9540P Cycles				
Panel	Pretreatment	10	20	40	60	80
1	Abrasive Blast Only	8	7	4	2	0
2		8	7	4	2	0
3		8	5	3	1	0
1	Dip Zn Phos/Cr6+ Gardobond 24T + FH-3	8	6	5	3	2
2		8	7	5	4	2
3		7	6	5	2	0
1	Spray Zn Phos + SurTec 580 Gardobond 24S + SurTec 580	7	7	6	3	3
2		7	7	5	5	4
3		7	6	5	5	5
1	Spray Zn Phos + CS100 Gardobond 24S + CS100	8	7	5	4	3
2		8	7	7	5	3
3		8	8	7	5	3

Good Bad

LCS Panels in GM9540P

Performance Objectives
rating ≥ 4 @ 60 cycles

- Alternatives met Performance Objectives
- Chromated Zn Phosphate did not meet objectives

After 60 hours GM9540P

Dip Zn Phos/Cr6+	Spray Zn Phos/SurTec 580	Spray Zn Phos/PPG Chemseal 100

TECHNOLOGY DRIVEN. WARFIGHTER FOCUSED.

Low Carbon Steel Panels Scraped after 80 Cycles GM9540P

Dip Zn Phos/Cr6+

Spray Zn Phos/SurTec 580

Spray Zn Phos/PPG Chemseal 100

Laboratory Validation Results

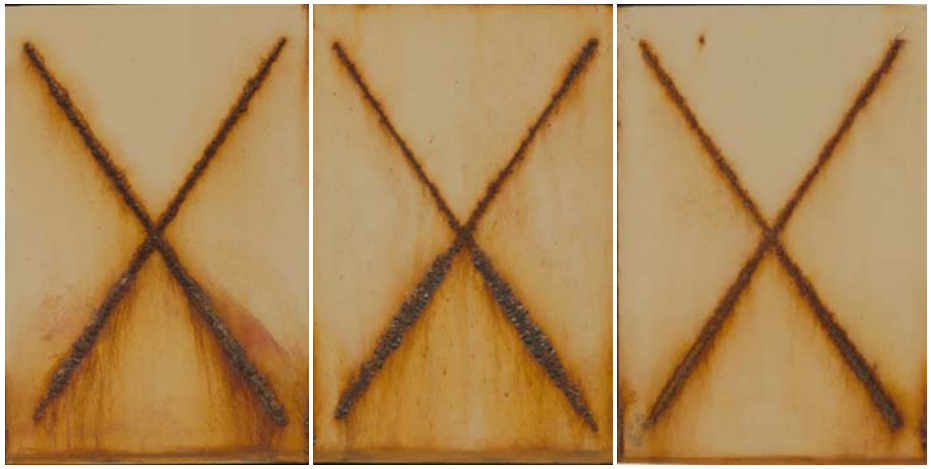
ASTM-D1654 Ratings for High Hard Armor (HHA) with 53022/53039						
		GM 9540P Cycles				
Panel	Pretreatment	10	20	40	60	80
1	Abrasive Blast Only	7	6	4	2	0
2		8	7	4	0	0
3		8	6	5	2	0
1	Dip Zn Phos/Cr6+ Gardobond 24T + FH-3	8	8	6	6	4
2		7	7	6	6	4
3		7	7	6	6	5
1	Spray Zn Phos + SurTec 580 Gardobond 24S + SurTec 580	8	7	5	5	3
2		7	7	6	4	2
3		6	6	5	5	5
1	Spray Zn Phos + CS100 Gardobond 24S + CS100	8	7	6	6	5
2		7	7	6	5	3
3		8	8	7	6	5

Good ● ▶ Bad

HHA Panels in GM9540P

Performance Objectives
rating ≥ 4 @ 60 cycles

HHA Panels after 60 hours GM9540P



Dip Zn Phos/Cr6+	Spray Zn Phos/SurTec 580	Spray Zn Phos/PPG Chemseal 100

TECHNOLOGY DRIVEN. WARFIGHTER FOCUSED.

HHA Panels Scraped after 80 Cycles GM9540P

Dip Zn Phos/Cr6+	Spray Zn Phos/SurTec 580	Spray Zn Phos/PPG Chemseal 100

Laboratory Validation Results

Chip Resistance SAE-J400 Gravelometer

Performance Objective NLT 5B

Milled Finish Low Carbon Steel						
	53022/53039		53022/64159		23377/85285 ZVOC	
Abrasive Blasted Only	4	B	4	B	5	B
Dip Zinc Phosphate/Cr6+	5	B	4	B	4	B
Spray Zinc Phos/Surtec 580	4	B	4	B	6	B
Spray Zinc Phos/CS100	5	B/A	5	B/A	5	B

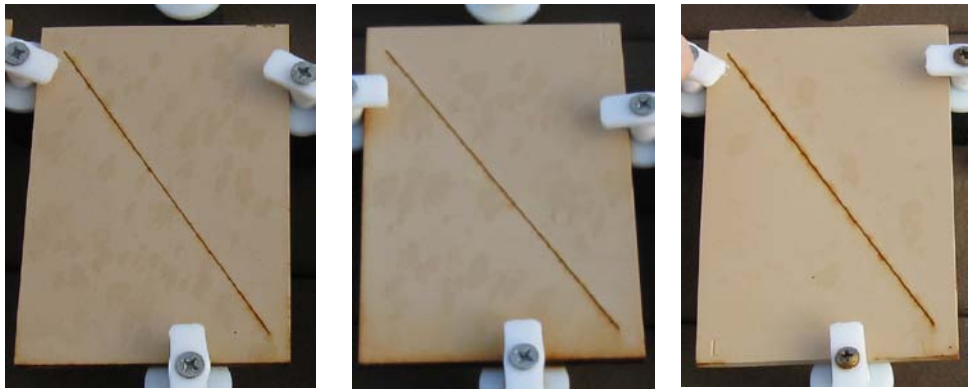
Abrasive Blasted High Hard Armor						
	53022/53039		53022/64159		23377/85285 ZVOC	
Abrasive Blasted Only	6	A/B	5	B/A	5	B
Dip Zinc Phosphate/Cr6+	5	B	5	B/A	5	B/A
Spray Zinc Phos/Surtec 580	5	B	4	B/A	5	B/A
Spray Zinc Phos/CS100	4	B/A	4	B	5	B

Laboratory Validation Results

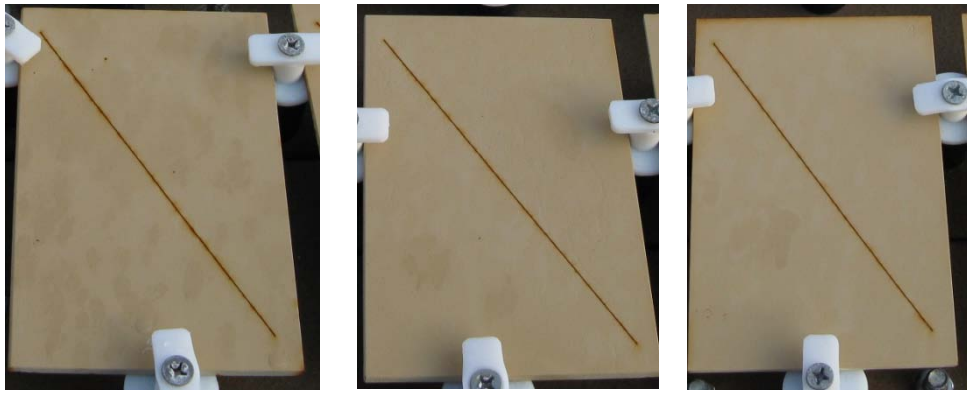
6 Months Outdoor Exposure at Cape Canaveral (December 2011)

- Shown with MIL-DTL-53022/MIL-DTL-53039 coating system
- SurTec 580 and CS100 performing well vs. chromate sealer

Low Carbon Steel



High Hard Armor



Dip Zn Phos/Cr6+	Spray Zn Phos/SurTec 580	Spray Zn Phos/CS 100

Laboratory Validation Summary

- Both SurTec 580 and PPG Chemseal 100 met the JTP Screening Requirements for demonstration
- Progress towards meeting Performance Objectives are mixed: (*Field tests and outdoor exposure remaining)*
  - SurTec 580 performed slightly better than Chemseal 100 on LCS in GM9540P, Chemseal better on HHA
  - Chromated Zn Phosphate failed GM9540P on LCS
- Both alternative sealers holding up well over 6 months outdoor exposure
- ***SurTec 580 selected for demonstration based on results to date and NAVAIR experience with TCP***

Demonstration

SurTec 580 TCP

Clean and degrease

Abrasive blasting with appropriate grit. Alternatives to abrasive blasting may be used

Immersion in zinc phosphate.

Rinse in cold water.

Sealer: SurTec 580 (TCP) Ambient Temperature

Rinse in cold water

Dry with compressed hot air or centrifugal drier.

VS.

Chromic Acid

Clean and degrease

Abrasive blasting with appropriate grit. Alternatives to abrasive blasting may be used

Immersion in zinc phosphate.

Rinse in cold water.

Sealer: 4 oz chromic acid, 4 oz phosphoric acid per 100 gal of solution at a temperature of 150-200°F.

Dry with compressed hot air or centrifugal drier.

Demonstration

450 Gal vat used for SurTec 580

SurTec 580 Demonstration

- Initiated at ANAD April 23, 2012
- Trackable M1 Components:
  - Fuel Caps
  - Copula
- Other misc parts and test panels

M1 Copula during SurTec 580 process

M1 Fuel Caps and test panels

Demonstration

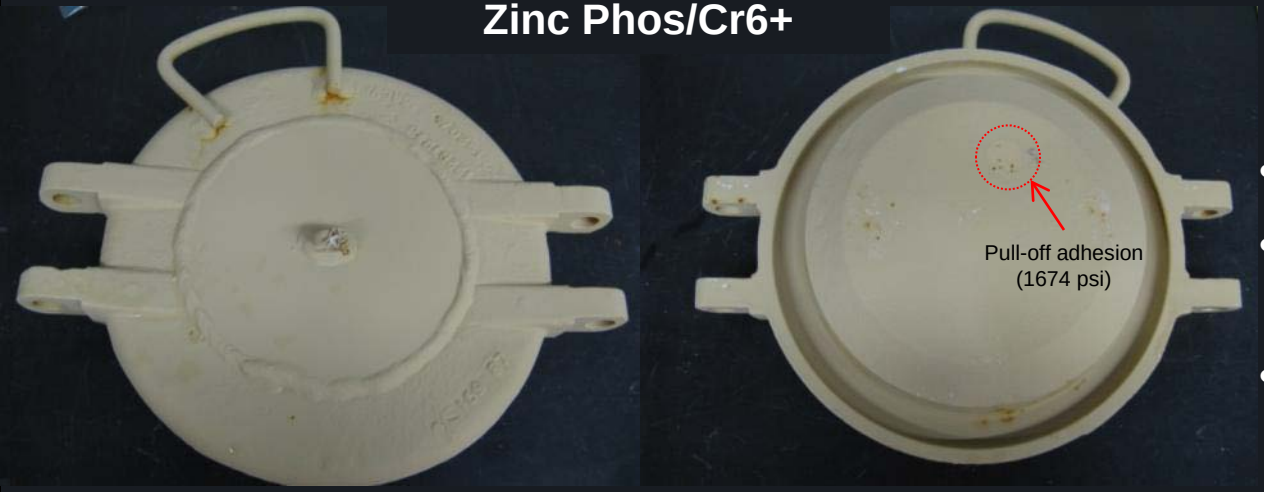
Demonstration of Non-Chromate Sealer Samples and Parts

QTY	Substrate	Sealer	Testing Facility
4	Steel (ANAD)	SurTec 580	ANAD
4	Steel (ANAD)	Chromate	ANAD
4	Steel (ANAD)	SurTec 580	ARL
4	Steel (ANAD)	Chromate	ARL
3	HHA	SurTec 580	ARL
2	HHA	Chromate	ARL
3	LCS	SurTec 580	ARL
2	LCS	Chromate	ARL
1	Steel U-Weld	SurTec 580	ARL
1	Steel U-Weld	Chromate	ARL
1	Fuel Cap	SurTec 580	ARL
1	Fuel Cap	Chromate	ARL
5	Fuel Cap	SurTec 580	Field
5	Fuel Cap	Chromate	Field
2	Copula	SurTec 580	Field

Demonstration

SurTec 580 Demonstration

Zinc Phos/Cr6+



Zinc Phos/SurTec 580

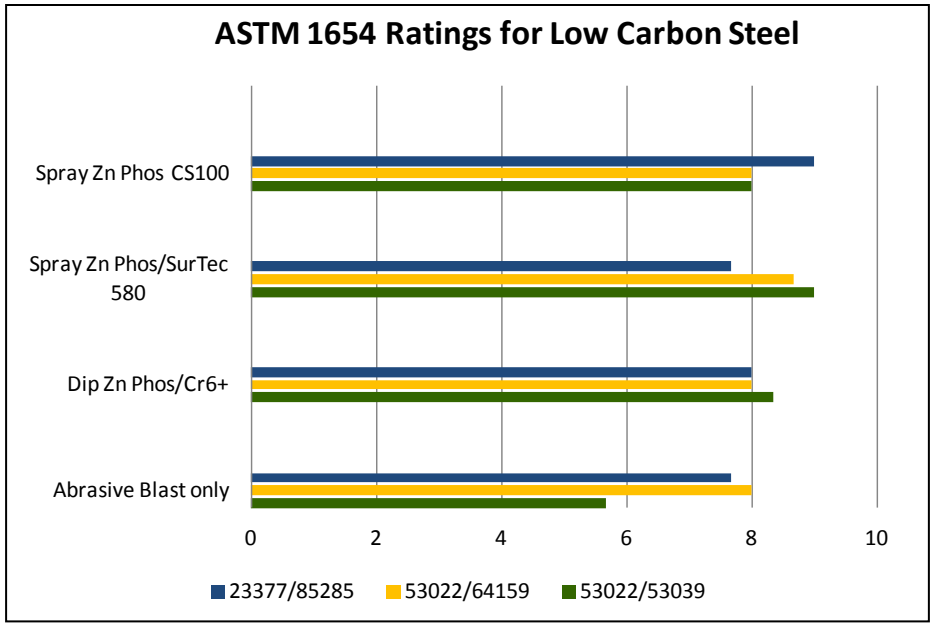
Demonstration Parts in GM9540P

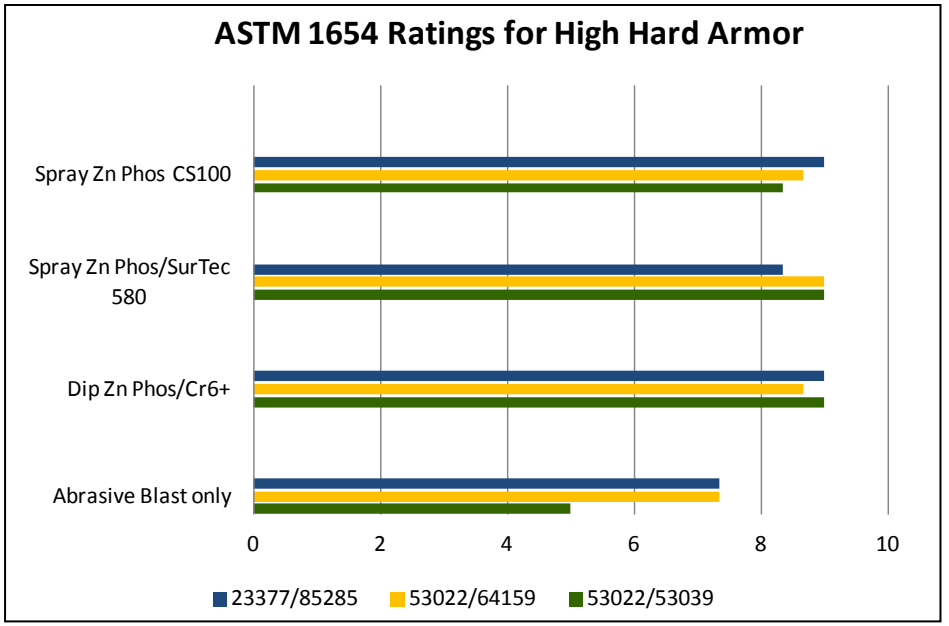
- Parts coated with
- MIL-DTL-53022/MIL-DTL-53039
- Similar performance after 20 cycles GM9540P
- Pull off adhesion nearly identical

Laboratory Validation Results

12 Months Outdoor Exposure at Cape Canaveral (June 2012)

- SurTec 580, and CS100 continue to perform well vs. chromate sealer
- Abrasive blast only (DTM) panels beginning to tail off.



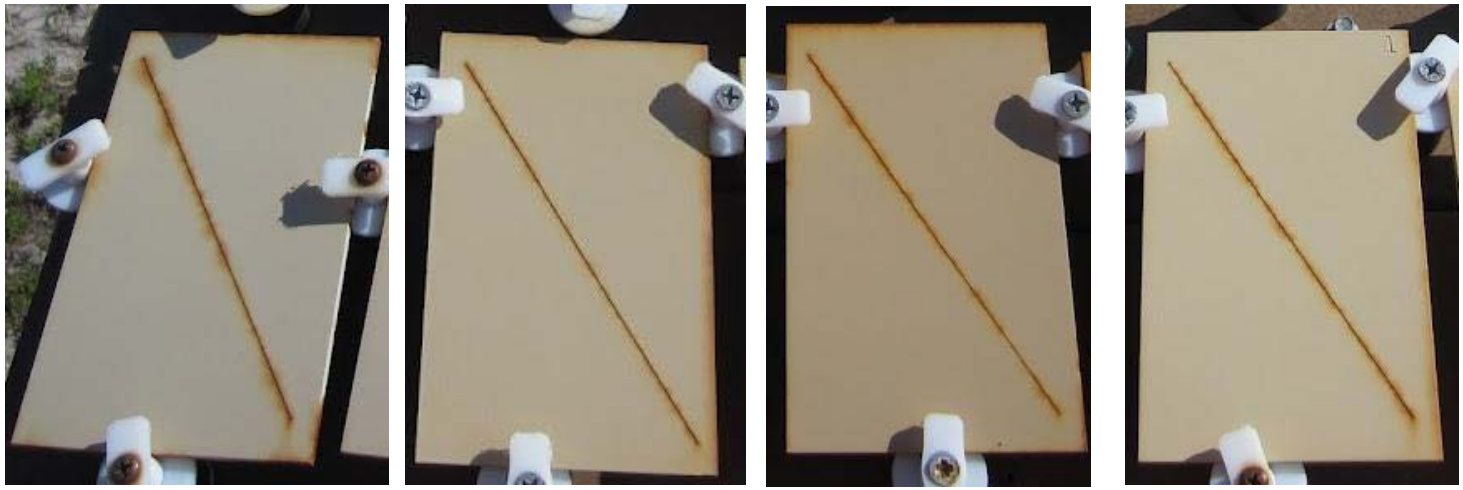


Laboratory Validation Results

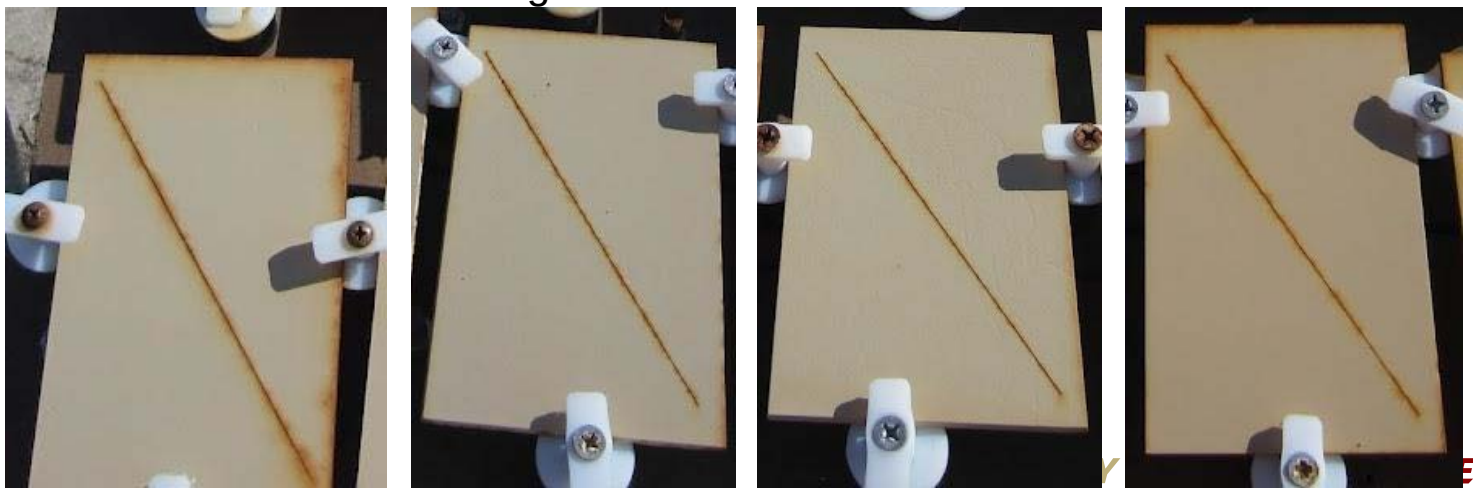
12 Months Outdoor Exposure at Cape Canaveral

Low Carbon Steel Panels

Abrasive Blast | Dip Phos/Cr6+ | Spray Phos/ ST580 | Spray Phos/ CS100



High Hard Armor Steel Panels



Summary

- SurTec 580 and PPG Chemseal 100 are proving to be viable alternatives to chromic acid rinse for zinc phosphate